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DMIC review OF RECENT DEVELOPMENTS

...ickel- and Cobalt-Base Alloys

...rennan, and W. F. Simmons April 8, 1970

ALLOY DEVELOPMENT

Turbine-Blade Alloy For Thin Sections

The development program of a turbine-blade casting alloy with improved ductility in thin sections has continued at TRW.(1) To date, superalloy air-cooled blades have characteristically exhibited poor intermediate-temperature ductility in thin sections.

The program was designed to permit a statistical treatment of the data. The first series of alloys were evaluated on the basis of the 1400 F tensile ductility in thin sections and creep strength at 1800 F. TRW NASA VI A alloy was chosen over U-700, IN-100, and IN-792 as the base composition for use in the second series of compositions. The TRW alloy will be coated to provide the necessary oxidation resistance.

Eight alloying additions (chromium, hafnium, tantalum, tungsten, aluminum, titanium, molybdenum, and vanadium) have been chosen for evaluation in the next part of the program.

Melting and Casting Procedures Developed for Nitinol

Suitable melting, casting, and working procedures for the production of commercial-size heats of 55-Nitinol have been developed at Battelle/Columbus.(2) This alloy (55 wt % Ni-45 wt % Ti) exhibits a rare ability to restore itself to a predetermined shape after being plastically deformed. The restoration is accomplished by reheating to a certain transition temperature. The transition temperature, a function of alloy composition, can range from below room temperature to above 250 F. The alloy also possesses other unusual properties below its transition temperature, e.g., a very high damping capacity. The alloy has a very low damping capacity above its transition temperature.

Applications likely to utilize the unusual properties of 55-Nitinol include: temperature-sensitive switches, mechanical-work devices, blind fasteners, and self-erectable antennas for aerospace applications.

Production of Large-Diameter, Close-Tolerance Castings

A program is being conducted by TRW to develop the capability of producing complex structural castings having large thin-walled areas.(3) Alloy 718 was selected as the material for evaluation since it is typical of the alloys with the required high-temperature mechanical properties for structural members of gas turbines. A diffuser case was selected as the casting for this program because it presents potential problems anticipated in casting structural components. TRW has designed and constructed the gating system of the mold for the diffuser-case casting. Also, wax patterns for making the mold have been assembled. TRW expects to cast and evaluate the first full diffuser case made of Alloy 718.

DISPERSION-STRENGTHENED ALLOYS

TD Nickel-Chromium Tubing

A program is being conducted by Fansteel to evaluate two processes to manufacture high-quality TD Nickel-Chromium tubing having a 2000 F ultimate tensile strength of about 15 ksi.(4) This tensile strength at 2000 F is now exhibited by TD Nickel-Chromium sheet. The goal sizes for tubing are 0.3125-inch OD x 0.035-inch wall, 0.250-inch OD x 0.020-inch wall, and 0.0156-inch OD x 0.020-inch wall.

The two processes being evaluated are (1) warm drawing of small-diameter hollows and (2) direct extrusion to about the final tubing size, followed by limited warm or cold drawing to the final size.

For the initial extrusion trials, graphite-plugged billets and billets for extrusion over a mandrel were extruded successfully into tubing having outside diameters of from 0.3 to 0.8 inch, in lengths up to 2 feet. In both cases, the billets were canned on the ID and OD with mild steel.

The surfaces of most of the TD Nickel-Chromium tubing after removing the mild steel canning material was reportedly smooth. However, some difficulty was experienced in removing all of the steel canning material by pickling. This problem was attributed to chromium diffusion into the steel from the TD Nickel-Chromium alloy.

On the basis of results to date, both processes being evaluated appear to be capable of producing small-diameter TD Nickel-Chromium hollows for redrawing. Also, the plugged-billet extrusion process looks promising for direct extrusion to tubing. Finally, the use of a mandrel during

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extrusion of small tubing appears to be limited by the tensile failure of the mandrel.

Mechanical-property tests have not yet been conducted on the TD Nickel-Chromium tubing.

Structural Development for Dispersion-Strengthened Alloys

McDonnell Douglas Astronautics is working on a continuing project which was started in February 1967, to develop a capability for the structural utilization of second-generation dispersion-strengthened (DS) alloys for operation up to temperatures of 2400 F in future Air Force vehicles.(5) The objectives of the program are to investigate potential structural applications of DS alloys, to develop design data for structural components, to develop fabrication and assembly techniques, and to evaluate a DS-alloy structural assembly under simulated operational conditions.

A vertical fin of the FDL-5A lifting reentry vehicle was selected as the structural assembly. The design of the fin assembly has been completed. The surface panels will consist of single-faced, corrugation-stiffened designs utilizing 0.010-inch-thick Ni-20Cr-2ThO_2 corrugations and 0.015-inch-thick Ni-20Cr-2ThO_2 face sheets. Fabrication of some of these face panels has been started. Rivets and screws of Ni-20Cr-2ThO_2 will be used for assembly of the primary structure in areas where the temperature will exceed 1500 F.

Load fittings as well as other components of the structural test assembly also are made of Ni-20Cr-2ThO_2. Proof tests conducted on load fittings indicated that the margin of safety was low. Thus, the fitting was redesigned to increase the safety factor.

Thermal and stress analyses also were conducted on the structural test assembly.

Dispersion-Strengthened Cobalt Alloys for Turbine Vanes

Studies on dispersion-strengthened cobalt alloys for turbine-vane applications have been continued by Pratt & Whitney.(6) Two thoria-containing cobalt alloys, Co-20Ni-18Cr-2ThO_2 and Co-20Ni-30Cr-2ThO_2, are being evaluated for environments associated with those of an advanced aircraft engine. Specimens having an airfoil shape were prepared by precision extrusion for mechanical and corrosion tests and metallographic studies. Results of prolonged exposures at temperatures between 2000 and 2400 F indicated that both alloys were metallurgically stable after such exposure. The alloy, containing 18 percent chromium, exhibited higher strengths at room temperature and at 2000 F than did the alloy containing 30 percent chromium. On the other hand, creep-rupture strengths of these alloys were lower than previously reported. Also, the 30 percent chromium alloy exhibited higher creep-rupture strengths than did the lower chromium alloy, which was found to possess the higher creep-rupture strength in the earlier work.

The corrosion resistance of the TD cobalt alloys is being evaluated in isothermal oxidation erosion and cyclic hot-corrosion tests. The preliminary results of static and dynamic oxidation tests indicated that the oxidation resistance of the two cobalt alloys is about the same at 2000 F. However, at 2200 F, the alloy containing 30 percent chromium showed an appreciably lower weight loss rate than did the 18 percent chromium alloy. Hot-corrosion resistance is being determined on the airfoil specimens in a rig designed to simulate the turbine environment. The results of the tests after only 60 hours showed that the 18 percent chromium alloy initially gained weight up to 40 hours of exposure and then began to lose weight. On the other hand, the 30 percent chromium alloy showed a gradual weight loss immediately. Additional testing time is required to fully describe the hot-corrosion behavior of each alloy.

Dispersion-Strengthened Cobalt-Base Alloy Produced From an Aqueous Solution

An investigation was completed by Curtiss-Wright to develop a process for producing a cobalt-base dispersion-strengthened alloy from a flash-dried aqueous solution of metal salts containing colloidal ThO_2.(7) Various processing procedures were developed which resulted in the production of of several lots of Co-18Cr-20Ni-4 vol % ThO_2 powder having a median ThO_2 particle diameter of 195 A. Procedures also were developed for processing the powders into extruded rods with approximately 100 percent of theoretical density. The average and median diameters of the ThO_2 particles in an extruded rod having the best combination of microstructural characteristics after a heat treatment at 2200 F for 100 hours were 1470 and 210 A, respectively.

CRACKING IN SUPERALLOYS

Research has recently begun at Lockheed-Georgia to develop procedures for reducing strain-age cracking of René 41 alloy weldments.(8) This program involves (1) the development of a standard crack-susceptibility test, (2) the evaluation of techniques for "desensitizing" René 41 weldments (by welding parameter manipulation and postweld thermal processing), and (3) the determination of the applicability of promising procedures developed for "desensitizing" René 41 weldments to "desensitize" other crack-sensitive nickel-base superalloys.

An acoustic-emission technique for remote monitoring of crack events during heat treatment was developed. In this technique, the acoustic energy of crack initiation and propagation in a modified "circular-patch restraint" specimen can be measured and observed on an oscilloscope as a function of test temperature. Test results showed that the technique is capable of discriminating the René 41 heat-treat cracking problem and that the data are reproducible. The acoustically defined cracking temperature was shown to be a crack-susceptibility test parameter that is responsive to variations in base metals, heat-treating procedures, and welding-process-parameter manipulations.

Strain-Age Cracking of René 41 Alloy

The strain-age cracking and mechanical-property reliability of René 41 was studied by Rocketdyne for F-1 rocket-engine applications.(9) René 41 was chosen for most of the turbine manifold because of its superior combination of fabricability and creep strength in the 1300 to 1700 F range, but strain-age cracking was encountered during manifold fabrication.

The study showed that controlled-heating-rate tensile tests and weld-circle-patch tests successfully predicted strain-age cracking (SAC). Low tensile-ductility (at elevated temperatures), particularly less than 2 percent minimum elongation, was found to adversely affect the SAC sensitivity.

A preweld heat treatment and postweld heat treatment in an inert atmosphere were effective in the reduction of SAC. Increasing the postweld heating rate and decreasing the weld energy were also beneficial, but to a smaller extent. Slow cooling from the solution-annealing temperature (prior to aging) improved the ductility but cut the rupture life in half.

This study demonstrated that the SAC resistance and the toughness and ductility of René 41 can be improved by modifying the morphology of the grain-boundary carbides through heat treatment.

SUPERALLOY TUBING IN GAS-TURBINE ENGINES

In a survey of tubing applications in current and advanced engines, Pratt & Whitney found that the amount of external tubing of a large turbofan engine equals about 1 percent of the dry engine weight.(10) Therefore, except for military engines where every pound saved can be significant, the demand for weight-saving materials is considerably less than in other component applications, such as disks, shafts, and cases. Cost, prior experience, and availability are by far the more important selection criteria.

Table 1 shows the materials used, and a general basis for the selection of tubing for various applications.

TABLE 1. GENERAL BASIS FOR SELECTION OF TUBING MATERIALS

Thermal Expansion Desired	Service Temperature	Strength Required: Low	Medium	High
Low	Low	AISI 430	AISI 410	17-7PH
	High	Hastelloy N	Hastelloy C	--
Medium	Low	Inconel 600	Inconel X-750	Inconel 718
	High	Hastelloy X	Inconel X-750	Waspaloy
High	Low	AISI 321(347)	21-6-9[a]	21-6-9[a]
	High	AISI 321	21-6-9[a]	--

(a) Armco stainless steel, 21Cr-6Ni-9Mn-0.05C.

HOT CORROSION

Mechanisms

Research on the hot-corrosion mechanism of nickel-base superalloys at United Aircraft has recently been completed.(11)

This study utilized three commercial superalloys (B-1900, U-700, and Waspaloy) and five nickel binary alloys (Ni-5Cr, Ni-8Cr, Ni-13Cr, Ni-17Cr, and Ni-1Al) in an effort to delineate the mechanism of attack.

Oxidation tests and electrochemical-cell studies indicated that Na_2O (a reaction product of Na_2SO_4 and the substrate) breaks down the protective oxide scale and prevents its reformation. Results indicated that the rapid rates of oxidation associated with sulfidation are not due to preferential oxidation of chromium-rich sulfides, or oxidation of the nickel sulfide eutectic phase.

Hot corrosion was simulated by applying a saturated, aqueous solution of Na_2SO_4 onto the alloy surfaces, evaporating the water, and subsequently exposing the alloys in flowing oxygen at 1470 to 1830 F. A sulfidized structure, consisting of a zone of alloy matrix plus sulfides separating the unaffected alloy from the oxide scale, resulted after the Na_2SO_4 reacted with the alloy substrate. The protective nature of the scale was destroyed either by thermal cycling or by a Na_2O + alumina reaction. Alloys that do not form alumina-rich surface scales (such as Waspaloy) were not susceptible to the accelerated attack, but aluminum-containing alloys (such as B-1900) were quite susceptible. Oxides of tin and samarium, as well as chromium, were found to inhibit sulfidation.

Role of Rare-Earth Additions

A study of the effect of rare-earth additions in nickel-base superalloys on the hot-corrosion mechanism was recently completed at the General Electric Research and Development Center.(12)

Hot corrosion in aircraft jet engines is a combination of accelerated oxidation and sulfidation caused by a Na_2SO_4 film that forms on the superalloy first-stage and second-stage turbine parts operating in marine atmospheres. The attack is augmented by a combination of sulfur in the jet fuel and NaCl in the air. Previous observations at GE have shown that rare-earth additions reduce the rate of hot corrosion in nickel-base superalloys.

Rare-earth additions (as oxides rather than in metallic form) were found to reduce the extent of hot corrosion, apparently by reducing the amount of the sulfides formed. The rapid oxidation of Ni_3S_2 is thought to lead to severe hot corrosion. The rare-earth oxides (CeO_2, La_2O_3, and Gd_2O_3) reduce the amount of Ni_3S_2 formation by gettering the sulfur to form oxysulfides of the M_2O_2S type. Of the three oxides studied, CeO_2 and La_2O_3 exhibited nearly equal effectiveness, but Gd_2O_3 was much less effective.

If the rare-earth elements are effective only when added as oxides, then powder-metallurgy techniques should be developed to produce a superalloy containing appropriate oxides and adequate high-temperature strength.

Coatings to Reduce Hot Corrosion

Hot-corrosion tests of 39 coating-alloy combinations were made on six nickel- and two cobalt-base alloys for times up to 150 hours at 1650 and 1800 F for the nickel-base alloys, and 1800 and 2000 F for the cobalt-base alloys, in a high-velocity environment resulting from the combustion of JP-5 fuel in air and ingestion of 35 ppm of sea salt.(13) The alloys included René 41, Udimet 700, Alloy 713C, B-1900, IN-100, SEL-15, X-40, and WI-52. The coatings, which were identified with the alloys only by code, were from the following PWA 4?, ALPAK, CODEP, UC, MDC, and HI-15.

Under the test conditions, protection could be afforded by at least one of the coatings on the nickel-base alloys for 150 hours at 1650 F, and 100 hours at 1800 F. At 1800 F, the relatively thin coatings on the cobalt-base alloy exhibited poorer protection than did coated nickel-base alloys. At 2000 F, the coatings on WI-52 did not afford protec-

tion beyond 60 hours. Performance was only slightly better on the higher chromium X-40 alloy.

Hot-corrosion tests of the six nickel-base alloys, uncoated, rated them roughly as follows:

Very Poor	- B-1900, IN-100, SEL-15
Poor	- Alloy 713C
Good	- René 41, Udimet 700.

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